



# **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

## **Order Summary Report**

Order Number:	J110/0122					
Project Name:	WWTS - Biweekly					
Customer Name(s):	FGD WWTS					
Customan Address.	2405 Bina Hall Dd					
Customer Address:	3195 Pine Hall Rd					
	Mailcode: Belews Steam Station					
	Belews Creek, NC 28012					
Lab Contact:	Jason C Perkins	Phone:	980-875-5348			
Report Authorized By: (Signature)		Date:		7/29/2011		

### **Program Comments:**

FGD Bi Monthly

There was detectable Se in the filter blank sample. Sample results were all greater than 10 times the filter blank value. Data OK to report.

### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with an "X" or "1" indicate a deviation from the method quality system or quality control requirement. All results are reported on a dry weight basis unless otherwise noted.

#### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

## **Sample ID's & Descriptions:**

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2011015065	BELEWS	13-Jul-11 8:00 AM	T. Johnson	FGD Purge Eff
2011015066	BELEWS	13-Jul-11 8:00 AM	T. Johnson	EQ TANK EFF.
2011015067	BELEWS	13-Jul-11 8:00 AM	T. Johnson	BIOREACTOR 1 INF.
2011015068	BELEWS	13-Jul-11 8:00 AM	T. Johnson	BIOREACTOR 2 INF.
2011015069	BELEWS	13-Jul-11 8:00 AM	T. Johnson	BIOREACTOR 2 EFF.
2011015070	BELEWS	13-Jul-11 8:00 AM	T. Johnson	Trip Blank
2011015071	BELEWS	13-Jul-11 8:00 AM	T. Johnson	FILTER BLANK
2011015072	BELEWS	13-Jul-11 1:00 PM	DAVID MORRIS	BIOREACTOR 1 INF.
2011015073	BELEWS	13-Jul-11 1:00 PM	DAVID MORRIS	HG BLANK BIOREACTOR 1 INF.
2011015074	BELEWS	13-Jul-11 1:10 PM	DAVID MORRIS	BIOREACTOR 2 INF.
2011015075	BELEWS	13-Jul-11 1:10 PM	DAVID MORRIS	Hg Blk BioReactor 2 Inf
2011015076	BELEWS	13-Jul-11 1:05 PM	DAVID MORRIS	BIOREACTOR 2 EFF.
2011015077	BELEWS	13-Jul-11 1:05 PM	DAVID MORRIS	Hg Blk BioReactor 2 Eff

### **Technical Validation Review**

### **Checklist:**

		COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		<b>✓</b> Yes	□ No		
		All Results are less than the laboratory reporting lim	nits.	Yes	<b>✓</b> No		
		All laboratory QA/QC requirements are acceptable.		<b>✓</b> Yes	□ No		
		The Vendor Laboratories have been qualified by the Analytical Laboratory	Э	N/A			
Rep	ort S	Sections Included:					
	<b>✓</b> Jo	b Summary Report	✓ Sub-contr	acted Laborat	ory Results		
	<b>✓</b> Sa	ample Identification	☐ Customer	Specific Data	Sheets, Reports, & Documentation		
	<b>✓</b> Te	echnical Validation of Data Package	Customer Database Entries				
	<b>✓</b> Aı	nalytical Laboratory Certificate of Analysis	Test Case	e Narratives			
	☐ Aı	nalytical Laboratory QC Report	✓ Chain of 0	Custody			
			☐ Electronic	Data Delivera	able (EDD) Sent Separately		

Reviewed By: Mary Ann Ogle Date: 7/29/2011

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### Order # J11070122

Site: FGD Purge Eff Sample #: 2011015065

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst			
MERCURY (COLD VAPOR) IN WAT	<u>ER</u>									
Mercury (Hg)	175	ug/L		5	EPA 245.1	22-Jul-11 11:22	AGIBBS			
TOTAL RECOVERABLE METALS E	Y ICP									
Boron (B)	138	mg/L		0.5	EPA 200.7	27-Jul-11 12:48	DJSULL1			
DISSOLVED METALS BY ICP-MS										
Selenium (Se)	3330	ug/L		10	EPA 200.8	26-Jul-11 11:22	KRICHAR			
TOTAL RECOVERABLE METALS E	SY ICP-MS									
Arsenic (As)	236	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Chromium (Cr)	287	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Copper (Cu)	237	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Nickel (Ni)	358	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Selenium (Se)	4870	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:32	KRICHAR			
Zinc (Zn)	524	ug/L		20	EPA 200.8	19-Jul-11 12:32	KRICHAR			
SELENIUM SPECIATION										
Vendor Parameter	Complete	•			V_AS&C					
TOTAL DISSOLVED SOLIDS										
TDS	19000	mg/L		10	SM2540C	20-Jul-11 14:45	CLEEMAN			

Site: EQ TANK EFF. Sample #: 2011015066

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst					
MERCURY (COLD VAPOR)	) IN WATER											
Mercury (Hg)	166	ug/L		2.5	EPA 245.1	22-Jul-11 11:24	AGIBBS					
TOTAL RECOVERABLE METALS BY ICP												
Boron (B)	147	mg/L		0.5	EPA 200.7	27-Jul-11 12:52	DJSULL1					
DISSOLVED METALS BY I	CP-MS											
Selenium (Se)	2890	ug/L		10	EPA 200.8	26-Jul-11 11:06	KRICHAR					
TOTAL RECOVERABLE M	ETALS BY ICP-MS											
Arsenic (As)	223	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR					
Chromium (Cr)	277	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR					
Copper (Cu)	228	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR					
Nickel (Ni)	294	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR					

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### Order # J11070122

Site: EQ TANK EFF. Sample #: 2011015066

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS E	BY ICP-MS						
Selenium (Se)	4940	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:27	KRICHAR
Zinc (Zn)	448	ug/L		20	EPA 200.8	19-Jul-11 12:27	KRICHAR

Site: BIOREACTOR 1 INF. Sample #: 2011015067

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS B	SY ICP						
Boron (B)	147	mg/L		0.5	EPA 200.7	27-Jul-11 12:26	DJSULL1
DISSOLVED METALS BY ICP-MS							
Selenium (Se)	136	ug/L		10	EPA 200.8	26-Jul-11 10:44	KRICHAR
TOTAL RECOVERABLE METALS B	SY ICP-MS						
Arsenic (As)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Copper (Cu)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Nickel (Ni)	12.9	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Selenium (Se)	152	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:22	KRICHAR
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	19-Jul-11 12:22	KRICHAR
SELENIUM SPECIATION							
Vendor Parameter	Complete	)			V_AS&C		

Site: BIOREACTOR 2 INF. Sample #: 2011015068

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS E	BY ICP						
Boron (B)	147	mg/L		0.5	EPA 200.7	27-Jul-11 12:31	DJSULL1
TOTAL RECOVERABLE METALS E	BY ICP-MS						
Arsenic (As)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Copper (Cu)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Nickel (Ni)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Selenium (Se)	23.9	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:17	KRICHAR
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	19-Jul-11 12:17	KRICHAR

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### Order # J11070122

Site: BIOREACTOR 2 EFF. Sample #: 2011015069

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst					
MERCURY (COLD VAPOR) IN WAT	<u>TER</u>											
Mercury (Hg)	< 1	ug/L		1	EPA 245.1	22-Jul-11 11:27	AGIBBS					
TOTAL RECOVERABLE METALS BY ICP												
Boron (B)	156	mg/L		0.5	EPA 200.7	27-Jul-11 12:35	DJSULL1					
TOTAL RECOVERABLE METALS BY ICP-MS												
Arsenic (As)	< 5	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Chromium (Cr)	< 5	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Copper (Cu)	< 5	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Nickel (Ni)	< 5	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Selenium (Se)	6.15	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Silver (Ag)	< 5	ug/L		5	EPA 200.8	19-Jul-11 12:12	KRICHAR					
Zinc (Zn)	< 10	ug/L		10	EPA 200.8	19-Jul-11 12:12	KRICHAR					
SELENIUM SPECIATION												
Vendor Parameter	Complete	е			V_AS&C							

Site: Trip Blank Sample #: 2011015070

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst					
TOTAL RECOVERABLE METALS	BY ICP											
Boron (B)	< 0.05	mg/L		0.05	EPA 200.7	27-Jul-11 12:22	DJSULL1					
TOTAL RECOVERABLE METALS BY ICP-MS												
Arsenic (As)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Chromium (Cr)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Copper (Cu)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Nickel (Ni)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Selenium (Se)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Silver (Ag)	< 1	ug/L		1	EPA 200.8	19-Jul-11 12:08	KRICHAR					
Zinc (Zn)	< 2	ug/L		2	EPA 200.8	19-Jul-11 12:08	KRICHAR					
SELENIUM SPECIATION												
Vendor Parameter	Complet	е			V_AS&C							

Site: FILTER BLANK Sample #: 2011015071

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

**DISSOLVED METALS BY ICP-MS** 

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#### Order # J11070122

Site: FILTER BLANK Sample #: 2011015071

Collection Date: 13-Jul-11 8:00 AM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

**DISSOLVED METALS BY ICP-MS** 

Selenium (Se) 2.38 ug/L 2 EPA 200.8 26-Jul-11 10:34 KRICHAR

Site: BIOREACTOR 1 INF. Sample #: 2011015072

Collection Date: 13-Jul-11 1:00 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

**MERCURY 1631 - DISSOLVED** 

Vendor Parameter Complete V\_BRAND

Site: HG BLANK BIOREACTOR 1 INF. Sample #: 2011015073

Collection Date: 13-Jul-11 1:00 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

**MERCURY 1631** 

Vendor Parameter Complete V\_BRAND

MERCURY 1631 - DISSOLVED

Vendor Parameter Complete V\_BRAND

Site: BIOREACTOR 2 INF. Sample #: 2011015074

Collection Date: 13-Jul-11 1:10 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

MERCURY 1631 - DISSOLVED

Vendor Parameter Complete V\_BRAND

Site: Hg Blk BioReactor 2 Inf Sample #: 2011015075

Collection Date: 13-Jul-11 1:10 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

**MERCURY 1631 - DISSOLVED** 

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### Order # J11070122

Site: Hg Blk BioReactor 2 Inf Sample #: 2011015075

Collection Date: 13-Jul-11 1:10 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631 - DISSOLVED

Vendor Parameter Complete V\_BRAND

Site: BIOREACTOR 2 EFF. Sample #: 2011015076

Collection Date: 13-Jul-11 1:05 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

MERCURY 1631 - DISSOLVED

Vendor Parameter Complete V\_BRAND

Site: Hg Blk BioReactor 2 Eff Sample #: 2011015077

Collection Date: 13-Jul-11 1:05 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

MERCURY 1631 - DISSOLVED

Vendor Parameter Complete V\_BRAND



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

July 20, 2011

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews – FGD (WWTS Bi-Monthly Sampling) (LIMS # J11070122)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on July 14, 2011. The samples were received in a sealed cooler at -0.4°C on July 15, 2011. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Ben Wozniak Project Manager

Ben Wozniek

Applied Speciation and Consulting, LLC

### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078

Project: Belews – FGD (WWTS Bi-Monthly Sampling) (LIMS # J11070122)

July 20, 2011

### 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on July 14, 2011. The samples were received on July 15, 2011 in a sealed container at -0.4°C.

The samples were received in a laminar flow clean hood void of trace metals contamination and ultra-violet radiation. Upon reception, the samples were designated discrete sample identifiers. An aliquot of each sample was filtered (0.45µm) and these filtrates were stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of

each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> All samples for selenium speciation analysis were analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on July 15, 2011. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

### 4. Analytical Issues

The overall analyses went very well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Ben Wozniak

Project Manager

Applied Speciation and Consulting, LLC

Ben Wozniek

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11070122

Date: July 20, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

### Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	211	103	ND (<3.4)	ND (<2.4)	ND (<2.4)	0 (0)
BioReactor 1 Inf	25.5	87.8	ND (<0.85)	1.06	ND (<0.59)	0 (0)
BioReactor 2 Eff	ND (<0.28)	ND (<0.64)	ND (<0.85)	ND (<0.59)	ND (<0.59)	0 (0)
Metals Trip Blk	ND (<0.055)	ND (<0.13)	ND (<0.17)	ND (<0.12)	ND (<0.12)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS n = number of unknown Se species observed

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11070122

Date: July 20, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

### **Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.006	0.055	0.28	1.1
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.013	0.13	0.64	2.6
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.017	0.17	0.85	3.4
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.012	0.12	0.59	2.4
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.012	0.12	0.59	2.4

eMDL = Estimated Method Detection Limit

### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	10.16	106.2
Se(VI)	LCS	9.48	9.786	103.2
SeCN	LCS	8.92	8.875	99.5
MeSe(IV)	LCS	6.47	6.446	99.6
SeMe	LCS	9.32	9.379	100.6

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD (WWTS Bi-Monthly Sampling) Contact: Jay Perkins LIMS #J11070122

Date: July 20, 2011 Report Generated by: Ben Wozniak Applied Speciation and Consulting, LLC

### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	BioReactor 2 Eff	ND (<0.28)	ND (<0.28)	NC	NC
Se(VI)	BioReactor 2 Eff	ND (<0.64)	ND (<0.64)	NC	NC
SeCN	BioReactor 2 Eff	ND (<0.85)	ND (<0.85)	NC	NC
MeSe(IV)	BioReactor 2 Eff	ND (<0.59)	ND (<0.59)	NC	NC
SeMe	BioReactor 2 Eff	ND (<0.59)	ND (<0.59)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

### Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	BioReactor 2 Eff	278.0	270.2	97.2	278.0	268.7	96.7	0.5
Se(VI)	BioReactor 2 Eff	252.3	239.7	95.0	252.3	239.3	94.9	0.2
SeCN	BioReactor 2 Eff	228.8	205.6	89.9	228.8	205.2	89.7	0.2

Du En	ike ergy₅	Duke Energy A  Mail Code MG 13339 H	nalytical Laboratory O3A2 (Building 7405) lagers Ferry Rd ille, N.C. 28078			70127MAT	rical Lal	oorat	ory		Only Sample Origina From	s		Page 16	of 2	7	ige 1 RIBU	TION
1)Project Name		(70	4) 875-5245 704) 875-4349 2)Phone No:	Logged By	œu .	7-14	-1/	//	80	7	SAM: Water	PLE PI		AM Ground NPDES Drinking Wate UST	1 1	COPY		
		lonthly Sampling)		AS	&C		Cool	cr Ten	nn IC	,		RCRA	A Was		-			
2) Client:	Bill Kennedy	y, Melonie Martin, an, Tom Johnson *	4)Fax No:	V PO	#13324	<u>t</u> )	15Prese 2=H <sub>2</sub> SO 4=Ice	rv.:1= 4 3=H	HCL INO <sub>3</sub>		3,4	4	3,4		T	4		
5)Business Unit:		6)Process:	Mail Code:	MR#				10	, k				*			2	s)	
B)Oper. Unit:		9)Res. Type:	10)Reso. Center:			o complete on-shaded a		16 Analyses	Required				e (no dig.)**			tion - vendor to	(Important to place filled back into both baggies)	
LAB USE ONLY	Se Speciation Bot	22000	e Description or ID	Sampling	Time	2nd and 4th We		17Сотр.	18 Grab	(0)	Hg - 245.1	Metals*	Se, soluble				AS&C (Import bottle back in	
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July 21, 2011

Duke Energy ATTN: Jay Perkins Scientific Support-Laboratory 13339 Hagers Ferry Road Huntersville NC 28078 jcperkins@duke-energy.com labcustomer@duke-energy.com

RE: Project DUK-HV1101 Client Project: J11070122

Dear Mr. Perkins,

On July 15, 2011, Brooks Rand Labs (BRL) received three (3) flue gas desulfurization (FGD) waste water samples and three (3) corresponding field blanks. Samples were logged-in for total mercury (Hg) analysis and were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the applicable SOP(s) and may be evaluated using adjusted reporting limits to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific detection limits and other details. All quality assurance criteria were satisfied, and all data was reported without additional qualification, aside from concentration qualifiers.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report. Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com

tilwate

Lydia Greaves
Jr. Project Manager
lydia@brooksrand.com

**S** 

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater

## Report Information

### **Laboratory Accreditation**

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

### **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	Т	total recoverable fraction

### **Definition of Data Qualifiers**

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- J-M Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- M Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.</u>

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



Analytical Laboratory Page 19 of 27

> Client PM: Jay Perkins Client PO: 141391

# Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1129042-01	FGD Wastewater	Sample	07/13/2011	07/15/2011
Hg Blk BioReactor 1 Inf	1129042-02	DIW	Field Blank	07/13/2011	07/15/2011
BioReactor 2 Inf	1129042-03	FGD Wastewater	QC Sample	07/13/2011	07/15/2011
Hg Blk BioReactor 2 Inf	1129042-04	DIW	Field Blank	07/13/2011	07/15/2011
BioReactor 2 Eff	1129042-05	FGD Wastewater	Sample	07/13/2011	07/15/2011
Hg Blk BioReactor 2 Eff	1129042-06	DIW	Field Blank	07/13/2011	07/15/2011

# **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	07/18/2011	07/20/2011	B111039	1100496



Project ID: DUK-HV1101

PM: Tiffany Stilwater

# Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1129042-01	<b>f</b> Hg	FGD Wastewater	Т	82.7		3.03	8.08	ng/L	B111039	1100496
<b>BioReactor 2 E</b> 1129042-05	<b>ff</b> Hg	FGD Wastewater	Т	24.5		0.77	2.05	ng/L	B111039	1100496
<b>BioReactor 2 In</b> 1129042-03	<b>f</b> Hg	FGD Wastewater	Т	65.4		3.03	8.08	ng/L	B111039	1100496
Hg Blk BioRead 1129042-02	c <b>tor 1 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B111039	1100496
<b>Hg Blk BioRead</b> 1129042-06	ctor 2 Eff Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B111039	1100496
<b>Hg Blk BioRead</b> 1129042-04	ctor 2 Inf Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B111039	1100496

BROOKS RAND LABS

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater

# Accuracy & Precision Summary

Batch: B111039 Lab Matrix: Water Method: EPA 1631

Sample	Analyte	Native	Spike	Result	Units	REC 8	Limits	RPD & Limits
B111039-SRM1	Certified Reference Materi	al (1128019,	NIST 1641d 1	l000x diluti	on)			
	Hg	•	15.68	15.39	ng/L	98%	85-115	
B111039-MS3	Matrix Spike (1129042-03)							
	Hg	65.36	323.2	429.8	ng/L	113%	71-125	
B111039-MSD3	Matrix Spike Duplicate (11	29042-03)						
	Hg	65.36	323.2	411.2	ng/L	107%	71-125	4% 24

# Method Blanks & Reporting Limits

Batch: B111039 Matrix: Water Method: EPA 1631 Analyte: Hg

Sample	Result	Units
B111039-BLK1	0.08	ng/L
B111039-BLK2	0.10	ng/L
B111039-BLK3	0.07	ng/L
B111039-BLK4	0.05	ng/L

 Average: 0.08
 Standard Deviation: 0.02
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.41

RAND LABS

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater

# **Instrument Calibration**

Sequence: 1100496 Total Mercury Speciation by CVAFS

Method: EPA 1631

Instrument: THG-05 Date: 07/20/2011 Analyte: Hg

Lab ID	True Value	Result	Units	REC	& Limits
1100496-IBL1		9.55	pg of Hg		
1100496-IBL2		9.37	pg of Hg		
1100496-IBL3		8.12	pg of Hg		
1100496-IBL4		9.29	pg of Hg		
1100496-CAL1	25.00	22.79	pg of Hg	91%	
1100496-CAL2	100.0	100.3	pg of Hg	100%	
1100496-CAL3	500.0	511.3	pg of Hg	102%	
1100496-CAL4	2500	2648	pg of Hg	106%	
1100496-CAL5	10000	10170	pg of Hg	102%	
1100496-ICV1	1568	1539	pg of Hg	98%	85-115
1100496-CCB1		14.7	pg of Hg		
1100496-CCV1	500.0	509.8	pg of Hg	102%	77-123
1100496-CCV2	500.0	507.1	pg of Hg	101%	77-123

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



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> Client PM: Jay Perkins Client PO: 141391

## Sample Containers

Lab ID: 1129042-01 Report Matrix: FGD Wastewater Collected: 07/13/2011 Sample: BioReactor 1 Inf Sample Type: Sample Received: 07/15/2011 Des Container **Size** Lot **Preservation** P-Lot Ship. Cont. Bottle FLPE Hg-T 125 mL No Lot# None N/A Cooler Lab ID: 1129042-02 Report Matrix: DIW Collected: 07/13/2011 Sample: Hg Blk BioReactor 1 Inf Sample Type: Field Blank Received: 07/15/2011 Des Container **Preservation** P-Lot Ship. Cont. Size Lot Bottle FLPE Hg-T 125 mL No Lot# None N/A Cooler Lab ID: 1129042-03 Report Matrix: FGD Wastewater Collected: 07/13/2011 Sample: BioReactor 2 Inf Received: 07/15/2011 Sample Type: QC Sample **Preservation** Container Size Lot P-Lot рΗ Ship. Cont. Bottle FLPE Hg-T 250 mL 71313080 None N/A Cooler 60 Comments: About 100 mL Sample Volume Lab ID: 1129042-04 Collected: 07/13/2011 Report Matrix: DIW Sample: Hg Blk BioReactor 2 Inf Received: 07/15/2011 Sample Type: Field Blank Des Container Preservation **Size** P-Lot pН Ship. Cont. Lot Bottle FLPE Hg-T 125 mL No Lot# None N/A Cooler Lab ID: 1129042-05 Report Matrix: FGD Wastewater Collected: 07/13/2011 Sample: BioReactor 2 Eff Sample Type: Sample Received: 07/15/2011 Container P-Lot Ship. Cont. Des Size Lot Preservation Bottle FLPE Hq-T 125 mL No Lot# None N/A Cooler Lab ID: 1129042-06 Report Matrix: DIW Collected: 07/13/2011 Sample: Hg Blk BioReactor 2 Eff Sample Type: Field Blank Received: 07/15/2011 Des Container Size Lot **Preservation** P-Lot Ship. Cont. Bottle FLPE Hg-T 125 mL No Lot# None N/A Cooler

**Project ID:** DUK-HV1101 **PM:** Tiffany Stilwater



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Client PM: Jay Perkins Client PO: 141391

# **Shipping Containers**

Cooler

**Received:** July 15, 2011 8:45

**Tracking No:** 4726 7966 2152 via FedEx

Coolant Type: Ice Temperature: 1.4 °C Description: Cooler
Damaged in transit? No
Returned to client? No

Custody seals present? No Custody seals intact? No COC present? Yes

CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM Analytical Laboratory Page 25 of 27 **Analytical Laboratory Use Only Duke Energy Analytical Laboratory** <sup>19</sup>Page 2 of **2** Duke ORDER# Sample Class OTHER Mail Code MGO3A2 (Building 7405) Originating DISTRIBUTION **Energy**... 13339 Hagers Ferry Rd From ORIGINAL to LAB Huntersville, N. C. 28078 SAMPLE PROGRAM COPY to CLIENT (704) 875-5245 Fax: (704) 875-4349 Drinking Water 1)Project Name Belews - FGD 2)Phone No: UST\_ RCRA Waste **Brooks Rand** WWTS (2011, Bi-Weekly Sampling) Cooler Temp (C) 2) Client: 4)Fax No: PO#141391 Bill Kennedy, Melonie Martin, 2=H,SO, 3=HNO. Wayne Chapman, Tom Johnson \* 4=ice 5=None 5 6)Process: 5)Business Unit: Mail Code: 2nd week 10)Reso. Center: 8)Oper. Unit: 9)Res. Type: Customer to complete all appropriate non-shaded areas. Hg 1631 (sample only) LAB USE ONLY 17Comp. 18 Grab Se Speciation Bottle ID <sup>13</sup>Sample Description or ID 11Lab ID Time Signature 2011015072 7/3-11 1300 BioReactor 1 Inf Hg Blk BioReactor 1 Inf 1300 BioReactor 2 Inf Hg Blk BioReactor 2 Inf BioReactor 2 Eff 1305 Hg Blk BioReactor 2 Eff Use the Bioreactor 2 Inf or EFF sample as the MS/MSD Customer to sign & date below - fill out from left to right, 1) Relinquished By 2) Accepted By desired turnaround. <sup>22</sup>Requested Turnaround 3) Relinguished 4) Accepted By Date/Time Customer, IMPORTANTI e indicate desired turnar 0845 5)Relinguished 6)Accepted B Date/Time 7)Relinquished By Date/Time 8)Accepted By: Date/Time 9)Seal/Locked/By 10) Seal/Lock Opened By Date/Time 11)Seal/Locked By 12)Seal/Lock Opened By Date/Time Please i Comments \* Metals=As, Ag, B, Cu, Cr, Ni, Se, Zn \*thomas.d.johnson@siemens.com

1129042

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